PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Process for the Production of Jellies or Viscous Solutions

We, MACLEANS LIMITED, a company organised under the laws of Great Britain, of Great West Road, Brentford, Middlesex, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention comprises a process for the production of jellies or viscous solutions which may be used as hand jellies, stabilizers, detergent builders, vehicles for medicaments, and for other purposes.

According to the invention, a process for the production of jellies or viscous solutions comprises mixing together at least two different carbohydrate complexes or derivatives at an elevated temperature in an aqueous medium, the carbohydrate complexes or derivatives being selected from agar-agar, carrageenin (Chondrus Extract), sodium carboxy methyl cellulose, or gum tragacanth. Preferably the mixing is conducted at a temperature within the range of 45—105° C.

The carbohydrate complexes or derivatives may be sodium carboxy methyl cellulose and carrageenin (Chondrus Extract) in which case one part by weight of the former is preferably employed for every 2—6 parts by weight of the latter. Alternatively, the carbohydrate complexes or derivatives may be agar-agar and gum tragacanth in which case four parts by weight of the former is preferably employed for every 5—9 parts by weight of the latter. Agar-agar itself forms a jelly of somewhat short texture which, when used as a hand jelly, breaks up into fragments which cannot be rubbed satisfactorily into the hands. When it is used in an aqueous solution at a low concentration, a short textured jelly is formed which rapidly exudes liquid at room temperatures (syneresis), and which becomes almost completely fluid at about 65° C. When it is employed in an aqueous solution at a high concentration, a jelly which does not liquefy up to about 85. C., may be obtained, but such a jelly is so tough at ordinary temperatures that it cannot be readily squeezed from tubes, is somewhat opaque and has poor light transmission properties. Agar-agar jellies, in common with those of most other gel forming materials, tend to lose their gelling power on continued exposure to heat.

Sodium carboxy methyl cellulose in aqueous solution will not of itself form a jelly although it is able to form viscous solutions.

When agar-agar and sodium carboxy methyl cellulose are dispersed in water and heated together the first effect is a degredation of the agar-agar with a complete loss of gelling power. On continued heating a reaction, which may be a polymerisation, takes place with progressively increasing development of gelling power. The mixture remains fluid while hot and may be maintained fluid at 50° C. for a considerable period. On cooling to 40° C. however, a jelly is formed. It is found that a heating period of from 4 to 7 hours at 80° C., or from 2 to 4 hours at 90° C., gives jellies of a suitable texture for use as hand jellies, while a longer heating period, or heating at 100° C., for periods of from a quarter of an hour to one hour, gives very tough gels.

We have noted, however, that if agar-agar and sodium carboxy methyl cellulose are dispersed separately in water and are heated to prepare solutions which are then mixed together, the reaction proceeds more quickly than if these materials are mixed cold and subsequently heated together. When the method of mixing together hot solutions of these reacting

materials is employed, the strength of the resultant jelly depends more upon the temperature of mixing than upon the duration of the heating, although the duration is of importance. Exact control over the strength and texture of the resulting jelly may be achieved by reserving a portion of the water which is to be employed in the jelly for use as a cold quench, the quench being added to the reacting solutions after they have been mixed at the desired temperature requsite to obtain the desired gel structure.

The invention is illustrated by the following examples. In Examples II—IV the amount of water needed will depend on what strength the resultant gel is required to possess.

EXAMPLE I.

A hand jelly is prepared having the following percentage (by weight) composition:—

Agar-agar - - - -	0.7
Sodium Carboxy Methyl Cellulose	1.4
Gum Tragacanth - - -	0.5
Glycerine - - -	8.0
Perfume, colour and preservative - -	q.s.
Water to - - - -	100

Agar-agar, and gum tragacanth are carbohydrate complexes.

The sodium carboxy methyl cellulose may be that manufactured under the Registered Trade Mark "Cellofas B—medium grade."

The agar-agar and gum tragacanth are mixed with half the glycerine and one third of the total water employed. The resulting mixture is heated to prepare a solution at 90° C. A solution at 90° C., is also prepared from the sodium carboxy methyl cellulose, the remaining glycerine and another third of the water. The two solutions are then mixed and the remaining water is added at 20° C. The resulting solution is allowed to cool in an enclosed tank to 45° C. when the perfume, colour and preservative are added. The solution is then kept at 42° C. to 43° C. until required for filling into tubes or pots.

When the reacting materials employed are such as to give a final composition of about 0.8% of agar-agar, and about 1.6% of sodium carboxy methyl cellulose, together with a glycerine content of about 4%, it is found that if the mixing temperature is in the range of 50° C. to 60° C., a low viscocity solution is obtained, but with a mixing temperature of 70° C. a high viscocity solution is obtained. When the mixing temperature is within the range 80° C. to 90° C. a medium textured jelly is obtained, whilst with a mixing temperature of 100° C. a strong jelly of somewhat short texture is obtained.

EXAMPLE II

Agar-agar and sodium carboxy methyl cellulose are mixed in aqueous solution at 50° C., one part by weight of agar-agar being employed for every two parts by weight of sodium carboxy methyl cellulose. A solution of very low viscosity is obtained. When solutions of these materials are mixed at 80 to 90° C. a medium texture gel is produced. When the mixing temperature is 100° C. a strong gel of somewhat short texture is obtained. A suitable jelly may be prepared containing 0.7% by weight of agar-agar and 1.4% by weight of sodium carboxy methyl cellulose.

EXAMPLE III

Carrageenin (Chondrus Extract) and sodium carboxy methyl cellulose are mixed in aqueous solution at 90° C., four parts by weight of the former being employed for every one part by weight of the latter. The mixture is maintained at 90° C. for several hours and a soft textured gel having good temperature characteristics, results. In the absence of the heating period the resultant gel liquefies at quite low temperatures. It has been found that a suitable jelly may be made containing 1.8% by weight of carrageenin (powder extract) and 0.45% by weight of sodium carboxy methyl cellulose. The reaction in this case is very much slower and the jelly produced softens more easily than is the case with a comparable jelly made from agar-agar and sodium carboxy methyl cellulose

EXAMPLE IV

Agar-agar and gum tragacanth, are reacted in aqueous solution at 50° C., about four parts by weight of agar-agar being employed for every seven parts by weight of gum tragacanth. A thin gel is produced. When the same materials are reacted in aqueous solutions at 90° C., a firmer gel of tougher texture is obtained. It has been found that a suitable jelly may be made containing 0.86% by weight of agar-agar and 1.44% by weight of gum tragacanth.

In performing the present invention the proportions by weight of the reacting materials employed may vary widely. Thus when it is desired to prepare a jelly by using as reacting materials sodium carboxy methyl cellulose and agar-agar from about half to ten times as much by weight of the former as of the latter may be used. The greater the proportion of the former the longer is the texture of the resulting jelly. The preferred propor-

tion, however, is one part by weight of agar-agar to two parts by weight of sodium carboxy methyl cellulose.

The proportion of the reaction product in the final gel may be of the order of 2—3%, but stronger gels may be obtained by using less water in the reaction. The lower the proportion of the reaction product in the final gel the greater is the tendency to syneresis, but this can be reduced substantially by small additions of certain hydrophilic colloids, for example sodium alginate and/or by the addition of certain electrolytes, for example soluble calcium salts.

There are listed below the characteristics of various jellies and viscous solutions which may be prepared in accordance with the invention.

Use	Characteristics
Hand jelly	Clarity; good temperature characteristics; freedom from stickiness, good texture emolliency; impression of being well "absorbed".
Stabiliser for tooth pastes, paste shampoos, shaving cream, and pastes and creams in general.	Favourable temperature characteristics,
Detergent builder and suspension agent for use with soaps and synthetic detergents.	Strong gelling power.
Suspension jelly for canned food products.	Enhances foaming and detergent power and possesses valuable thickening effect.
Vehicle for medicaments.	Strong gel with good suspending power and temperature stability.
Surgical lubricant.	Can be sterilised and is emollient and non-lathering; wide range of compatibilities.
Culture medium in bacteriology.	Can be sterilised; emollient.
Bulk laxative.	Clarity; capable of being sterilised; probable specificity.
Hair setting lotions, creams and other toilet preparations.	Water retention powers; non-toxic nature.
Thickener and/or gelling agent for ice-cream, jams, jellies and other food products.	Low solids content with strong structure.
	Flavourless; "setting" easy to control; good temperature characteristics.

We are aware that it has previously been proposed to stabilize ice-cream without appreciably raising its viscosity by the addition of sodium carboxy-methyl-cellulose together with a relatively small quantity of carrageenin. This differs essentially from the present invention which is concerned with producing jellies and viscous solutions. In carrying out the present invention when sodium carboxy-methyl-cellulose and carrageenin are used together it is necessary that the amount by weight of carrageenin should exceed that of sodium carboxy-methyl-cellulose.

What we claim is:—

1. A process for the production of jellies or viscous solutions comprising mixing together at least two different carbohydrate complexes or derivatives at an elevated temperature in an aqueous medium, the carbohydrate complexes or derivatives being selected from agar-agar, carrageenin (Chondrus Extract), sodium carboxy methyl cellulose, or gum tragacanth.

2. A process as claimed in Claim 1 in which the mixing is conducted at a temperature within the range of 45—105° C.

3. A process as claimed in Claim 1 in which the carbohydrate complexes or derivatives are sodium carboxy methyl cellulose and agar-agar.

4. A process as claimed in Claim 3, in which from 0.5 to 10 parts by weight of sodium carboxy methyl cellulose are employed for every part of agar-agar.

5. A process as claimed in Claim 1 in which the carbohydrate complexes or derivatives are carrageenin (Chondrus Extract) and sodium carboxy methyl cellulose.

6. A process as claimed in Claim 5 in which one part by weight of sodium carboxy methyl cellulose is employed for every 2—6 parts by weight of carrageenin.

7. A process as claimed in Claim 1 in which the carbohydrate complexes or derivatives are agar-agar and gum tragacanth.

8. A process as claimed in Claim 7 in which four parts by weight of agar-agar are employed for every 5—9 parts by weight of gum tragacanth.

9. A process as claimed in any of the preceding claims in which the proportion of the reaction product in the final gel is substantially 2—3%.

10. A process as claimed in any of the preceding claims in which the final gel comprises a hydrophilic colloid and/or an electrolyte.

11. A process for the production of jellies or viscous solutions substantially as described in any of the examples.

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PROVISIONAL SPECIFICATION
No. 3753, A.D. 1952.

Process for the Production of Jellies or Viscous Solutions

We, MACLEANS LIMITED, a Company organised under the laws of Great Britain, of Great West Road, Brentford, Middlesex, do hereby declare this invention to be described in the following statement:—

This invention comprises a process for the production of jellies which may be used as hand jellies, stabilizers, detergent builders, vehicles for medicaments, and for other purposes.

According to the invention a process for the production of jellies comprises reacting together agar-agar or carrageenin (Chondrus Extract) with sodium carboxy methyl cellulose and/or gum tragacanth at an elevated temperature in an aqueous medium.

Agar-agar itself forms a jelly of somewhat short texture which, when used as a hand jelly, breaks up into fragments which cannot be rubbed satisfactorily into the hands. When it is used in an aqueous solution at a low concentration, a short textured jelly is formed which rapidly exudes liquid at room temperatures (syneresis), and which becomes almost completely fluid at about 75° C. When it is employed in an aqueous solution at a high concentration, a jelly which does not liquefy up to about 85° C. may be obtained, but such a jelly is so tough at ordinary temperatures that it cannot be readily squeezed from tubes, is somewhat opaque and has poor light transmission properties. Agar-agar jellies, in common with those of most other gel forming materials, tend to lose their gelling power on continued exposure to heat.

Sodium carboxy methyl cellulose in aqueous solution will not of itself form a jelly although it is able to form viscous solutions.

In performing the present invention the proportions, by weight of the reacting materials employed may vary widely.

Thus where the reacting materials are sodium carboxy methyl cellulose and agar-agar, from about half to ten times as much of the former as the latter may be used. The greater the proportion of the former the longer is the texture of the resulting jelly. The preferred proportion, however, is one part by weight of agar-agar to two parts by weight of sodium carboxy methyl cellulose.

Where the reacting materials employed are carrageenin and sodium carboxy methyl cellulose the preferred proportion is four parts by weight of the former to one part by weight of the latter. It has been found that a suitable jelly may be made containing 1.8% by weight of carrageenin (powder extract) and 0.45% by weight of sodium carboxy methyl cellulose. The reaction in this case is very much slower and the jelly produced softens more easily at raised temperatures than is the case with a comparable jelly made from agar-agar and sodium carboxy methyl cellulose.

Where the reacting materials employed are agar-agar and gum tragacanth, the preferred proportion is about 4 parts by weight of agar-agar to 7 parts by weight of gum tragacanth. It has been found that a suitable jelly may be made containing 0.86% by weight of agar-agar and 1.44% by weight of gum tragacanth. Where these reacting materials are mixed together at 50° C. a thin gel is produced, while mixing of these materials at 90° C. produces a firmer gel of tougher texture

The proportion of the reaction product in the final gel may be of the order of 2—3%, but stronger gels may be obtained by using less water in the reaction. The lower the proportion of the reaction product in the final gel the greater is the tendency to syneresis, but this can be reduced substantially by small additions of certain hydrophilic colloids, for example gum tragacanth or sodium alginate, and/or by the addition of certain electrolytes, for example soluble calcium salts.

When agar-agar and sodium carboxy methyl cellulose are dispersed in water and heated together the first effect is a degradation of the agar-agar with a complete loss of gelling power. On continued heating a reaction, which may be

a polymerisation, takes place with progressively increasing development of gelling power. The mixture remains fluid while hot and may be maintained fluid at 50° C. for a considerable period. On cooling to 40° C. however a jelly is formed. It is found that a heating period of from 4 to 7 hours at 80° C., or from 2 to 4 hours at 90° C., gives jellies of a suitable texture for use as hand jellies, while a longer heating period, or heating at 100° C. for periods of from a quarter of an hour to one hour, gives very tough gels.

We have noted, however, that if agar-agar and sodium carboxy methyl cellulose are dispersed separately in water and are heated to prepare solutions which are then mixed together, the reaction proceeds more quickly than if these materials are mixed cold and subsequently heated together. When the method of mixing together hot solutions of these reacting materials is employed, the strength of the resultant jelly depends more upon the temperature of mixing than upon the duration of the heating, although the duration is of importance. Exact control over the strength and texture of the resulting jelly may be achieved by reserving a portion of the water which is to be employed in the jelly for use as a cold quench, the quench being added to the reacting solutions after they have been mixed at the temperature requisite to obtain the desired gel structure.

The invention is illustrated by the following example:—

A hand jelly is prepared having the following percentage (by weight) composition:—

Agar-agar	0.7
Sodium carboxy methyl cellulose	1.4
Gum tragacanth	0.5
Glycerine	8.0
Perfume, colour and preservative	q.s.
Water to	100

The sodium carboxy methyl cellulose may be that manufactured under the Registered Trade Mark "Cellofas B—medium grade".

The agar-agar and gum tragacanth are mixed with half the glycerine and one third of the total water employed. The resulting mixture is heated to prepare a solution at 90° C. A solution at 90° C. is also prepared from the sodium carboxy methyl cellulose, the remaining glycerine and another third of the water. The two solutions are then mixed and the remaining water is added at 20° C. The resulting solution is allowed to cool in an enclosed tank to 45° C. when the perfume, colour and preservative are added. The solution is then kept at 42° C. to 43° C. until required for filling into tubes or pots.

When the reacting materials employed are such as to give a final composition of about 0.8% of agar-agar, and about 1.6% of sodium carboxy methyl cellulose, together with a glycerine content of about 4%, it is found that if the mixing temperature is in the range 50° C. to 60° C., a low viscosity solution is obtained, but with a mixing temperature of 70° C. a high viscosity solution is obtained. When the mixing temperature is within the range 80° C. to 90° C. a medium textured jelly is obtained, whilst with a mixing temperature of 100° C. a strong jelly of somewhat short texture is obtained.

Gels produced in accordance with the invention have very good light transmission properties and are accordingly of good brilliance. The gels do not soften again until quite high temperatures are reached and, in the case of the tougher gels, some retention of structure is maintained even at 85 C. in spite of a low concentration of the reacting materials.

Since the materials employed are used in low concentrations, economical jellies are obtained. A gel of comparable strength made from agar-agar alone would require a very much higher concentration of agar-agar, and agar-agar is more expensive than sodium carboxy methyl cellulose.

There are listed below the characteristics of various jellies which may be prepared in accordance with the invention:—

Use	Characteristics
Hand jelly	Clarity; good temperature characteristics; freedom from stickiness, good texture, emolliency; impression of being well "absorbed".
Stabiliser for tooth pastes, paste shampoos, shaving cream, and pastes and creams in general.	Favourable temperature characteristics, Strong gelling power.
Detergent builder and suspension agent for use with soaps and synthetic detergents.	Enhances foaming and detergent power and possesses valuable thickening effect.
Suspension jelly for canned food products.	Strong gel with good suspending power and temperature stability.
Vehicle for medicaments.	Can be sterilised and is emollient and non-lathering; wide range of compatibilities.
Surgical lubricant.	Can be sterilised; emollient.
Culture medium in bacteriology.	Clarity; capable of being sterilised; probable specificity.
Bulk laxative.	Water retention powers; non-toxic nature.
Hair setting lotions, creams and other toilet preparations.	Low solids content with strong structure.
Thickener and/or gelling agent for ice-cream, jams, jellies and other food products.	Flavourless; "setting" easy to control; good temperature characteristics.

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PROVISIONAL SPECIFICATION
No. 8567, A.D. 1952.

Process for the Production of Jellies or Viscous Solutions

We, MACLEANS LIMITED, a Company organised under the laws of Great Britain, of Great West Road, Brentford, Middlesex, do hereby declare this invention to be described in the following statement:—

This invention comprises a process for the production of jellies and viscous solutions which may be used as hand jellies, stabilizers, detergent builders, vehicles for medicaments, and for other purposes. A process for the production of jellies of this type has been disclosed in our co-pending application No. 3753/52.

According to the invention, a process for the production of jellies or viscous solutions comprises reacting together at least two different carbohydrates at an elevated temperature in an aqueous medium. The carbohydrates may be compounds having metallic ions and/or acid radicals. Alternatively, the metallic ions and acid radicals are not present in the combined form in the molecules of the reacting carbohydrates but are introduced during the reaction. The reacting carbohydrates may be mono-, di-, or polysaccharides.

The invention is illustrated by the following examples in all of which the amount of water needed will depend on what strength the resultant gel is required to possess:—

(a) A monosaccharide, namely glucose, and a polysaccharide, namely agar-agar, are mixed in aqueous solution at 50° C., about seven parts by weight of glucose being employed for every four parts by weight of agar-agar. A very thin gel is produced. When glucose and agar-agar are mixed at 90° C., a gel of moderate strength and texture is obtained. A suitable jelly may be made containing 1.44% glucose and 0.86% agar-agar, the proportions being by weight.

(b) A disaccharide, namely sucrose, and a polysaccharide, namely agar-agar, are mixed in aqueous solution at 50° C., about seven parts by weight of sucrose being employed for every four parts by weight of agar-agar. A very thin gel is produced. When agar-agar and sucrose are mixed at 90° C. a gel of moderate strength and texture is obtained. A suitable jelly may be made containing 1.44% sucrose and 0.86% agar-agar, the proportions being by weight.

(c) Two materials containing polysaccharides, namely agar-agar and sodium carboxy methyl cellulose, are mixed in aqueous solution at 50° C., one part by weight of agar-agar being employed for every two parts by weight of sodium carboxy methyl cellulose. A solution of very low viscosity is obtained.

When solutions of these materials are mixed at 80 to 90° C., a medium texture gel is produced. When the mixing temperature is 100° C. a strong gel of somewhat short texture is obtained. A suitable jelly may be prepared containing 0.7% by weight of agar-agar and 1.4% by weight of sodium carboxy methyl cellulose.

(d) Two materials containing polysaccharides, namely carrageenin (Chondrus Extract) and sodium carboxy methyl cellulose, are mixed in aqueous solution at 90° C., four parts by weight of the former being employed for every one part by weight of the latter. The mixture is maintained at 90° C. for several hours and a soft textured gel, having good temperature characteristics, results. In the absence of the heating period the resultant gel liquefies at quite low temperatures. It has been found that a suitable jelly may be made containing 1.8% by weight of carrageenin (powder extract) and 0.45% by weight of sodium carboxy methyl cellulose. The reaction in this case is very much slower and the jelly produced softens more easily than is the case with a comparable jelly made from agar-agar and sodium carboxy methyl cellulose.

(e) Two materials containing polysaccharides, namely agar-agar and gum tragacanth, are reacted in aqueous solution at 50° C., about four parts by weight of agar-agar being employed for every seven parts by weight of gum tragacanth. A thin gel is produced. When the same materials are reacted in aqueous solution at 90° C., a firmer gel of tougher texture is obtained. It has been found that a suitable jelly may be made containing 0.86% by weight of agar-agar and 1.44% by weight of gum tragacanth.

In performing the present invention the proportions by weight of the reacting materials employed may vary widely. Thus when it is desired to prepare a jelly by using as reacting materials sodium carboxy methyl cellulose and agar-agar from about half to ten times as much of the former as of the latter may be used. The greater the proportion of the former the longer is the texture of the resulting jelly. The preferred proportion, however, is one part by weight of agar-agar to two parts by weight of sodium carboxy methyl cellulose.

There are listed below the characteristics of various jellies and viscous solutions which may be prepared in accordance with the invention:—

Use	Characteristics
Hand jelly	Clarity; good temperature characteristics; freedom from stickiness, good texture, emolliency; impression of being well "absorbed".
Stabiliser for tooth pastes, paste shampoos, shaving cream, and pastes and creams in general.	Favourable temperature characteristics, Strong gelling power.
Detergent builder and suspension agent for use with soaps and synthetic detergents.	Enhances foaming and detergent power and possesses valuable thickening effect.
Suspension jelly for canned food products.	Strong gel with good suspending power and temperature stability.
Vehicle for medicaments.	Can be sterilised and is emollient and non-lathering; wide range of compatibilities.
Surgical lubricant.	Can be sterilised; emollient.
Culture medium in bacteriology.	Clarity; capable of being sterilised; probable specificity.
Bulk laxative.	Water retention powers; non-toxic nature.
Hair setting lotions, creams and other toilet preparations.	Low solids content with strong structure.
Thickener and/or gelling agent for ice-cream, jams, jellies and other food products.	Flavourless; "setting" easy to control; good temperature characteristics.

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